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(54) STEAM STRIPPING OF BLENDERS

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The present invention relates to a process for removing volatile impurities from polymeric material and particularly to a process for removing volatile hydrocarbon impurities from polymers of alpha-olefins made by a solution process.

With most polymerization reactions, the final processing step is usually one of recovering or obtaining the desired polymers in one form or another. The recovery of polymer may be handled in many ways, but in cases where the polymer has been made in solution, solvent removal assumes a major significance. In such cases, one of the more important criticalities of the recovered polymer is the level of residual solvent and other volatiles. This level must necessarily be low and preferably tend to zero to avoid serious problems through the tendency of the volatiles to produce a flammable or explosive concentration of vapours when stored in a confined space.

Solvent may be removed from polymer solution by suddenly dropping the pressure of the hot solution from above atmospheric to essentially atmospheric pressure whereby the major portion of the solvent flashes off, leaving between 0.5 and 5% by weight of the solvent in the polymer and then discharging the mixture of polymer solution and vaporized solvent into a receiver from which the vapour is withdrawn. For economical reasons and to eliminate prolonged heating of the polymer solutions, it is desirable that evaporation of the solvent and recovery of a polymer essentially completely free of volatiles be carried out in as few stages as possible. Once the solvent has been removed as far as possible, the polymer is extruded and frequently cut into pellets or other comminuted forms all hereinafter referred to as pellets. Regardless of how effectively the solvent has been removed, the polymer pellets usually retain some residual solvent and other volatiles

which must be removed before they can be further handled.

In the past, the residual solvent and other volatiles have been stripped from polymer pellets by passing hot air through a bed of such polymer pellets. Extreme caution is required when operating in this manner, since the presence of volatiles in hot air provides a fire hazard.

It is an object of the present invention to provide a method for stripping residual solvent and other volatiles from polymer by a method which is substantially free from fire hazard.

It has been found that the addition of steam at atmospheric pressures to air produces a safe stripping medium and, surprisingly, that such a stripping medium substantially reduces the time previously required for removing residual solvent and other volatiles from polymer pellets resulting from a solution polymerization process.

Accordingly, the present invention provides a process for removing residual volatile impurities from pellets of polymeric material resulting from a solution polymerization process, comprising the steps of subjecting a mass of such pellets to a gaseous mixture of steam and hot air, controlling the free oxygen concentration in the gaseous mixture to a concentration not exceeding 15% by volume of the mixture, passing the gaseous mixture through the mass of pellets and recovering pellets of polymeric material substantially free from volatile impurities.

The concentration of free oxygen in the gaseous mixture is preferably between 8% and 10% by volume of the mixture but may tend to zero if hot air is excluded.

According to one embodiment of the process of the present invention, the mass of pellets of polymeric material is agitated whilst the gaseous mixture passes through the mass. Such agitation may be accomplished by tumbling or stirring according to well-known procedures.

The process of the present invention may also be

practised simultaneously with the blending of pellets of polymeric material having different physical or chemical properties. Such blending is often desirable in order to reduce variations in product characteristics, to combine polymeric material from two or more different processes or production lines or to incorporate liquid or dry additives into the mass of polymeric material.

The gaseous mixture of steam and hot air is advantageously used at atmospheric pressure according to the process of the present invention. At such pressure, it is desirable to maintain the temperature of the gaseous mixture at above 75°C. to effect efficient stripping of the residual volatile impurities from the pellets. Temperatures in excess of 105°C. or much below 75°C. even at pressures other than atmospheric may not be desirable. Lower temperatures may be inefficient and higher temperatures may soften the polymeric material. Nonetheless, higher temperatures may be used as long as such higher temperatures do not cause the polymer pellets to fuse or stick together. In all cases where temperatures lower than 100°C. are employed at atmospheric pressure, the proportion of steam to air must be such that the air is capable of carrying essentially all of the steam as water vapour. The necessary amount of air to accomplish this may readily be calculated by well-known methods.

The removal of volatile impurities according to the present invention may be carried out with polyolefin polymers prepared by polymerization of monoolefinic aliphatic olefin monomers such as ethylene, propylene, or butylene or polymerizable mixtures thereof.

These polymers or copolymers of ethylene, propylene and other non-aromatic hydrocarbon olefins may be obtained under relatively low pressures using so-called coordination catalysts or catalysts such as chromium oxide on silicated alumina, hexavalent molybdenum compounds and charcoal-supported

nickel-cobalt. The coordination catalysts may be obtained by mixing a compound of, for example, titanium or zirconium, preferably one in which these metals are attached to groups such as -oxyhydrocarbon, -halides or any combination thereof, with an organometallic reducing agent as the second component. Examples of the second component are metal alkyls, alkenyls, acetylides such as lithium-aluminum ethyl cyclohexenyl, metal hydrides such as lithium hydride and sodium borohydride, Grignard re-agents such as phenol magnesium bromide, and alkali or alkaline earth metals such as sodium or lithium.

The polymer solutions are usually obtained by polymerizing the monomer or monomers in an inert solvent such as a hydrocarbon solvent which may be a paraffinic or aromatic hydrocarbon solvent such as hexane, cyclohexane, benzene, toluene or xylene. The polymerization reactor effluent usually contains polymer, solvent, unreacted monomer and catalyst. A normal sequence of processing may entail flashing off unreacted monomer followed by filtering out catalyst which leaves a solution of polymer in the solvent. This solution is then treated in such a way as to remove as much of the solvent as possible. After removal of the solvent, the polymer is usually extruded into strands and cut into pellets (or may be cut by a melt cutter). The pellets still contain some residual solvent and other volatile materials which must be removed before packaging for safety and processing considerations.

Pellets having the same or different physical or chemical properties may be treated in blending apparatus according to the process of the present invention. Typical blending apparatus could comprise a vertical cylindrical vessel with a conical base, a screw conveyer operating in a draft tube along the vertical axis of the vessel open at both ends, and an air-steam distributing system.

In such an arrangement, the material to be blended is conveyed by the screw upward from the cone apex, distributed in a layer on the top of the main body of material in the blending apparatus, and is then allowed to flow by gravity outside the draft tube to its starting point at the bottom of the blending apparatus. A mixture of air and steam is introduced into the cone through screened entrance ports from distributor rings around the outside of the cone. The air and steam are allowed to exhaust to atmosphere through weather protected openings on the top of the vessel.

The process of the present invention may also advantageously be used in connection with blending apparatus in which the polymeric material is conveyed by an exterior air lift. The mixing of the material is then accomplished by multiple takeoff points in the blending apparatus and steam is injected into the conveying air to form the gaseous mixture for removing the volatile impurities. The steam and air are exhausted to atmosphere through vents in the conical base of the blender.

The following Examples further illustrate the present invention. (All percentages are by volume unless otherwise shown.)

EXAMPLE 1

An experimental apparatus was constructed in such a way that pellets of a copolymer of ethylene with about 0.5% of butene, having a density of 0.949 gm/cc and a melt index of 0.65, contained in a glass tube 1.5 feet in length and 1 inch in diameter, could be treated with either air or steam or a mixture of air and steam. The gas or gaseous mixture was allowed to enter the top of the tube, permeate through the pellets of polymeric material and leave through the base of the tube. Prior to entering the tube, the gas or gaseous mixture was passed through a heat exchanger to ensure that its

temperature was at 100°C. The tube was insulated to maintain the temperature of the gas or gaseous mixture. A charge of about 50 grams of polymeric material was in the tube at any one time and gas flow rates were controlled by a rotameter. The results obtained with various gases or gaseous mixtures are tabulated below:

TABLE 1

Volatiles (Wt. %)

Time (Min.)	20% Oxygen (100% Air)	10% Oxygen (50% Air/ 50% Steam)	5% Oxygen (25% Air/ 75% Steam)	100% Steam
0	0.79	0.79	0.79	0.79
45	0.71	0.55	0.41	0.40
90	0.71	0.41	0.33	0.24

EXAMPLE II

Example I was repeated using pellets of a homopolymer of ethylene having a density of 0.960 gm/cc and a melt index of 13.5. The volatiles in wt. % remaining after treating the homopolymer with steam or air are tabulated below:

TABLE 2

Time (Min.)	100% Steam	100% Air
0	1.0	1.0
15	0.83	0.935
30	0.70	0.88
45	0.585	0.82
60	0.492	0.77
75	0.41	0.72
90	0.346	0.672
105	0.29	0.63
120	0.244	0.59
135	0.202	0.555
150	0.17	0.52

EXAMPLE III

Pellets of a homopolymer of ethylene having a density of 0.960 gm/cc, obtained by low-pressure polymerization of ethylene using a coordination catalyst, were introduced into a stainless steel, tubular blender, having a conically shaped base, of about 40,000 lbs. capacity. The following results were obtained after repeatedly filling the blender to capacity with the pellets and each time treating the latter with either air or a mixture of air and steam. For each run, pellets were chosen in such a way that the inital content of volatiles varied.

TABLE 3

Volatiles (Wt. %)

Time (Min.)	100% Air 1st	100% Air 2nd	10% Oxygen (50% Air/50% Steam) 3rd	10% Oxygen (50% Air/50% Steam) 4th run
0	0.45	0.82	1.20	0.33
60	0.35	0.59	0.33	0.19
120		0.50	0.07	0.12
180		0.40		
240				
300	0.18	0.23		

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for removing residual volatile impurities from pellets of polymeric material resulting from a solution polymerization process, comprising the steps of subjecting a mass of such pellets to a gaseous mixture of steam and hot air, controlling the free oxygen concentration in the gaseous mixture to a concentration not extending 15% by volume of the mixture, passing the gaseous mixture through the mass of pellets and recovering pellets of polymeric material substantially free from volatile impurities.

2. A process according to Claim 1 in which the polymeric material is a polymer of an alpha-olefin.

3. A process according to Claim 1 or Claim 2 in which the polymeric material is a homo- or co-polymer of ethylene.

4. A process according to Claim 1 in which the concentration of the free oxygen in the gaseous mixture is between 8% and 10% by volume of the mixture.

5. A process according to any one of Claims 1, 2, or 4 in which the gaseous mixture is passed through a mixture of pellets of polymeric material having different properties.

6. A process according to Claim 1 in which the temperature of the gaseous mixture is above 75°C.

7. A process according to Claim 6 in which the temperature of the gaseous mixture is above 75°C. and below 105°C.

8. A process according to any one of Claims 1, 4, or 7 which is carried out at atmospheric pressure.

SUBSTITUTE

REMPLACEMENT

SECTION is not Present

Cette Section est Absente